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MÜNCHEN LONDON

Translation of a Technical Report drawn up by crenox, R&D Labor

H · E File: 112 700 / cse

Test number: V 10/17	**Test starting date: 26 August 2010**
Ordering party: R&D, Dr. Auer	**Debited cost center:**
Order number:	
Test manager: Günnel	
	(signature)

Documentation: **CC: Auer**

LIDOS keywords: **filter salt**
composition
content of other metals
crystallization
building materials
conditioning

Purpose:

Production of conditioned filter salts with titanium contents of 3-20% relating to iron for examination in cements (background: patent).

Examination of the influence of the TiO_2 contents in the filter salt on chromate reduction.

Brief description:

A waste acid from the production is defined by the addition of $FeSO_4 \times 7H_2O$ (copperas) or $TiOSO_4$ solution to various Ti/Fe ratios.

Filter salt is crystallized from these waste acids by means of concentration in a rotary evaporator.

Subsequently, a partial neutralization (target: ~pH 4.0) of the adhering residual acid is carried out by mixing with limestone powder.

The preparations are used for technological examinations of concrete.

EXHIBIT 5

Conclusion:

The target values of the Ti/Fe ratios of 3/8/9.1/12/17/20% could be met approximately in the filter salt preparations.

In the end, 3.9/8.3/9.3/11.7/16.0/16.9% Ti/Fe were found, and therefore the highest Ti concentration was missed relatively clearly.

With increasing titanium content, the filter salt exhibited increased sulphuric acid contents (increasing from 15.8% to 23.0%).

The salts were processed further and partially neutralized by dry mixing with limestone powder, with the amount of said limestone powder addition increasing with the increasing acid content of the filter salt from a mass ratio of 1:0.20 (filter salt / limestone powder) to 1:0.30.

After storing for >4 days, DIN pH ranges from 1.9-2.2 (after 1 min stirring) and 4.2-4.6 (after three h stirring) were determined.

Educts:

- waste acid of 31 August 2010
- copperas from the production: average sample August 2010 (18.39% Fe)
- titanyl sulfate solution from the production of 31 August 2010 (8.84% TiO_2)
- Wuelfrath limestone powder 10/90

Analysis Waste Acid:

TiO_2 photom. [%]	$FeSO_4$ [%]	H_2SO_4 free [%]	**Ti/Fe** [%]	Al [g/l]	Ba [mg/l]	Ca [mg/l]	Cr [mg/l]	Na [mg/l]	Mg [g/l]	Mn [mg/l]	**Mn/Fe** [%]	Density 60°C [g/ml]
0.43	7.75	18.53	9.1	2.6	0.16	220	290	650	5.5	730	2.0	1.256

Preparations: In order to produce >400g of partially neutralized filter salt sample amounts with different TiO_2 contents, 2 crystallization trials with ~1.5kg starting solution (3 l flask) are necessary for each preparation:

Test	Ti / Fe (target) [%]	Provided amount waste acid [g]	Addition amount copperas [g]	Addition amount $TiOSO_4$ solution [g]
A1+A2	3	1500	469.1	0
B1+B2	8	1500	30.6	0
C1+C2	as is	1500	0	0
D1+D2	12	1500	0	23.7
E1+E2	17	1564.1	0	64.1
F1+F2	20	1500	0	88.2

Processing: An oil bath tempered to 90°C is used, with the concentration being carried out in one step (by a pressure reduction as steady as possible over a duration of approximately 4h to up to ~1mbar) and, subsequently, the crystal slurry being further agitated over night at 60°C (without pressure with 50 rpm at the rotavapor).

Subsequent filtration via jacket-heated pressure filter (60°C) (filter cloth: Markert PPD 3124).

Test	Total time [hours]	Total amount used (including $TiOSO_4$) [g]	Amount of evaporated water [g]	Weight thickened sludge [g]	Weight filter salt [g]	Weight pre-concentrated acid [g]	Total amount [filter salt + pre-concentrated acid] [g]
A1	4:36	1969,1	884,8	1069,8	574,4	438,8	1008,2
A2	4:09	1969,1	863,9	1094,5	556,7	461,4	1018,1
A_{ges}	*8:45*		*1748,7*	*2164,3*	*1131,1*	*900,2*	*2026,3*
A3	2:45		236,2	-236,2	119,5		
A_{ges}	11:30	3938,2	1984,9	1928,1	1011,6	462,5	1474,1
B1	4:54	1530,6	886,5	636,7	260,9	330,8	591,7
B2	4:23	1530,6	898,5	627,0	245,9	337,5	583,4
B_{ges}	9:17	3061,2	1765,0	1263,7	506,8	668,3	1175,1
C1	4:55	1500	833,6	654,8	225,0	395,3	620,3
C2	4:50	1500	907,2	590,1	235,3	320,6	555,9
C_{ges}	9:45	3000	1740,8	1244,9	460,3	715,9	1176,2
D1	4:40	1523,7	887,2	626,8	233,4	361,7	595,1
D2	4:50	1523,7	892,5	627,1	237,4	365,6	603,0
D_{ges}	9:30	3047,4	1779,7	1253,9	470,8	727,3	1198,1
E1	6:15	1628,2	915,6	707,5	248,7	427,9	676,6
E2	6:15	1628,2	935,8	694,1	242,6	415,3	657,9
E_{ges}	12:30	3256,4	1851,4	1401,6	491,3	843,2	1334,5
F1	5:21	1588,2	972,6	609,2	236,8	331,8	568,6
F2	4:28	1588,2	961,1	622,4	234,9	353,5	588,4
F_{ges}	9:49	3176,4	1933,7	1231,6	471,7	685,3	1157,0

ges = total

Analysis: After homogenization of the respective 2 preparations, these were subsequently analyzed.

Pre-Concentrated Acid:

No.	Density [g/ml]	TiO_2 photom. [%]	$FeSO_4$ [%]	H_2SO_4 free [%]
A	1.637	0.02	0.05	65.48
B	1.636	0.04	0.17	63.62
C	1.624	0.07	0.44	62.19
D	1.632	0.12	0.53	61.92
E	1.591	0.18	0.76	57.88
F	1.720	0.65	1.12	64.67

Filter Salt: Homogenization of the preparations 1+2 of each test

Nr.	TiO_2 photom. [%]	$FeSO_4$ [%]	H_2SO_4 frei [%]	Ti/Fe [%]	Al [g/kg]	Ba [mg/kg]	Ca [mg/kg]	Cr [mg/kg]	Na [g/kg]	Mg [g/kg]	Mn [g/kg]	Mn/Fe [%]
A	1,32	54,59	15,84	3,9	2,8	0,7	410	380	1,1	8,7	2,2	1:1
B	2,35	46,10	18,49	8,3	2,9	0,8	890	720	1,3	16,6	3,6	2:1
C	2,66	46,62	20,13	9,3	2,9	0,8	870	730	1,3	16,2	3,9	2:1
D	3,23	45,12	20,80	11,7	3,1	0,9	870	740	1,4	16,2	3,8	2:1
E	4,63	47,22	19,56	16,0	2,7	1,0	910	710	1,5	14,8	3,6	2:1
F	4,51	43,49	22,96	16,9	3,2	0,9	300	650	1,6	14,7	3,3	2:1

frei = free

Preliminary Test Regarding the Partial Neutralization of the Filter Salts:

3.0g each of the filter salt mixtures were dissolved in 27.0g of deionized water (= DIN pH), and Wuelfrath limestone powder 10/90 was added until a final pH value of ~4.0 was reached.

No.	DIN pH	Limestone powder [g]	Final pH
A	0.95	0.62	4.22
B	1.04	0.72	4.04
C	0.84	0.79	4.09
D	0.94	0.77	3.99
E	0.93	0.75	4.02
F	0.80	0.89	4.02

Partial Neutralization:

400g filter salt each are provided in the Krups-3-Mix, and x g of Wuelfrath limestone powder 10/90 were added and this was kneaded for 5 minutes. The mixtures are transferred into a poly beaker and stored (lid resting loosely on the beaker because of CO_2 generation).

After approx. 2d, the pH of the mixtures is measured (if there are large variations, it has to be corrected, if necessary, by admixture of filter salt or limestone powder).

No.	Amount filter salt [g]	Amount limestone powder [g]	Mass ratio filter salt/limestone powder	DIN ph after 2 min	DIN ph after 30 min
A	400	80,0	1:0,20	2,22	3,35
B	400	96,0	1:0,24	2,02	2,32
C	400	105,3	1:0,26	2,12	2,68
D	400	102,7	1:0,26	1,91	2,06
E	400	100,0	1:0,25	2,22	3,09
F	400	118,7	1:0,30	2,19	3,18

Final Products:

After storing for >4d, the pH values of the mixtures are determined after 1-180min stirring.

No.	Proportion filter salt [g]	Proportion limestone powder [g]	Mass ratio filter salt/ limestone powder	DIN ph after 1'	DIN ph after 5'	DIN ph after 15'	DIN ph after 30'	DIN ph after 60'	DIN ph after 180'
A	400	80,0	1:0,20	2,19	2,28	2,89	3,76	4,29	4,31
B	400	96,0	1:0,24	1,97	2,01	2,13	2,36	2,89	4,24
C	400	105,3	1:0,26	2,03	2,25	2,43	2,78	3,81	4,64
D	400	102,7	1:0,26	1,91	1,90	1,94	2,07	2,46	4,27
E	400	100,0	1:0,25	2,10	2,39	2,82	3,45	3,96	4,39
F	400	118,7	1:0,30	2,00	2,05	2,17	2,41	3,07	4,33

VERIFICATION

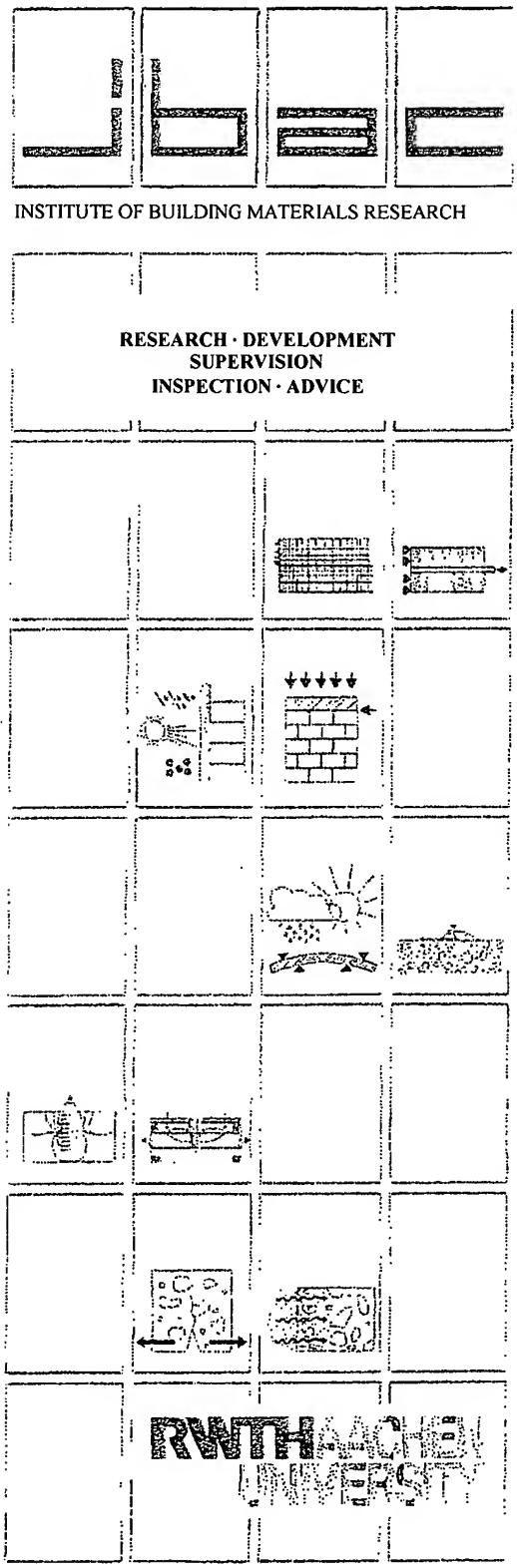
I, Dr. Gerhard Auer, hereby declare that all statements made in the Crenox Technical Report V 10/17, dated August 26, 2010 of my/our own knowledge are true, all statements made herein on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like are punishable by fine or imprisonment, or both, under 18 U.S.C. 1001, and may jeopardize the validity of the application or any patent issued thereon.

Date:

April 06, 2011

Dr. Gerhard Auer

Translation of the test report drawn up by IBAC Institut für Bauforschung Aachen



Test Report
F 7067/3

Determination of the efficiency and resistance of ferrous sulfate as a chromate reducing agent
- 3rd Interim Report -

EXHIBIT 6

RWTH RHEINISCH-WESTFÄLISCHE TECHNISCHE HOCHSCHULE AACHEN

ibac Institute of Building Materials Research Aachen

Ra/Sc
Second copy

SUBJECT

Determination of the efficiency and resistance of ferrous sulfate as a chromate reducing agent
- 3rd Interim Report –

No. of test report

F 7067/3
of 2 March 2011

Project management

Dr. rer. nat. R. Rankers

Contractor/ Promotor

crenox GmbH
Building N 304
Rheinuferstr. 7-9
47829 Krefeld

Date of order/order confirmation

25 March 2008

Reference number

-

This report consists of 3 pages, 3 of them with text.

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1 Procedure

crenox Pigments GmbH, Krefeld, appointed the Institute of Building Materials Research Aachen (ibac) to test the efficiency and resistance of ferrous sulfate as a chromate reducing agent (CR).

This report contains the results of tests carried out with 9 different chromate reducing agents (CR) delivered by the contractor in October and December 2010 to ibac. The efficiency was tested using the CEM I 52.5 N, which was made available by the contractor.

2 Preparation of samples

The delivered chromate reducing agents were carefully crushed in a mortar in order to guarantee homogeneity during the process of addition to the cement.

3 Laboratory tests – Examination of efficiency

First of all, the contents of ferrous ions in the different chromate reducing agents were determined and the respective stoichiometric amount of chromate reducing agents was calculated that is necessary to fully reduce the chromate content of the cement used for the further tests. To test the efficiency of the chromate reducing agents, the cement was mixed with the eight-fold stoichiometric amount.

The mixtures of cement and chromate reducing agents were homogenized by shaking for a time period of 24 hours. Subsequently, the chromate content of these mixtures was directly determined.

The determinations of the chromate values in all mixtures were performed photometrically according to DIN EN 196-10 /1/. The results are summarized in Table 1.

Table 1 Results of the determination of chromate according to DIN EN 196-10; determination directly after mixing the cement with the chromate reducing agent (eight-fold stoichiometric CR dosage)

Chromate reducing agents	CR dosage	chromate content of the cement mixture
	g CR/kg cement	mg/kg
1	2	3
none	-	13.0
V10/18 sample A (1)	2.1277	0.13
V10/18 sample B (2)	2.5381	1.11
V10/18 sample C (3)	2.7322	1.89
V10/17 sample A (7)	1.9231	0.33
V10/17 sample B (8)	2.2026	0.04
V10/17 sample C (9)	2.4631	0.04
V10/17 sample D (10)	2.3855	0.04
V10/17 sample E (11)	2.5227	0.04
V10/17 sample F (12)	2.6330	1.61

4 Literature

/1/ DIN EN 196-10:2006-10 Methods of testing cement, part 10: Determination of the water-soluble chromium (VI) content of cement

Head of Institute
(signature)
Dipl.-Ing. A. Vollpracht

Project management
(signature)
Dr.rer.nat. R. Rankers

VERIFICATION

We, Dr. R. Rankers and Dipl.-Ing. A. Vollpracht, hereby declare that all statements made in the Test Report F 7067/3 dated March 2, 2011, and entitled "Determination of the efficiency and resistance of ferrous sulfate as a chromate reducing agent" of my/our own knowledge are true, all statements made herein on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like are punishable by fine or imprisonment, or both, under 18 U.S.C. 1001, and may jeopardize the validity of the application or any patent issued thereon.

Date:

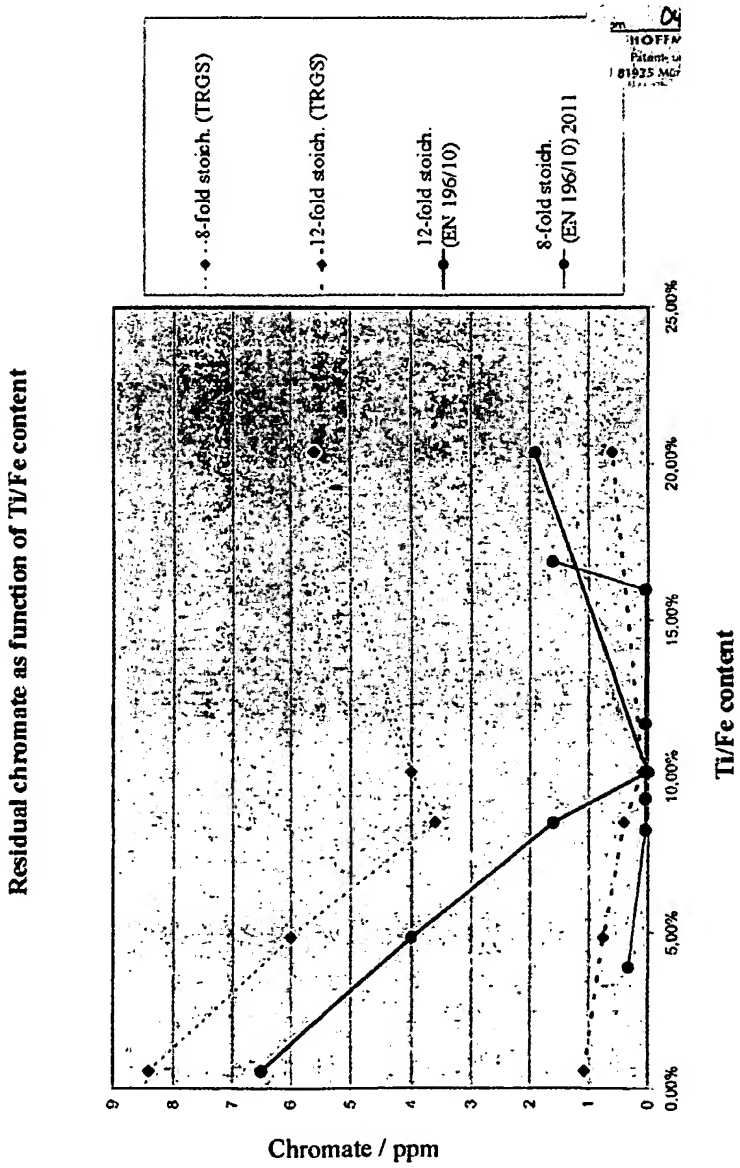
07.04.2011

Dipl.-Ing. A. Vollpracht

Dr. rer. nat. R. Rankers

Translation of Exhibit 55e

H · E File: 112 700 / cse



Technical Report F 7067/1	Table 1	"8-fold stoich. (TRGS)"
Technical Report F 7067/1	Table 1	"12-fold stoich. (TRGS)"
Technical Report F 7067/1	Table 2	"12-fold stoich. (EN 196/10)"
Technical Report F 7067/3	Table 1	"8-fold stoich.(EN 196/10) 2011"

EXHIBIT 7